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Investigation of the Ultrafiltration Technique Using Military Greases

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by
In-Sik Rhee

Report Date
October 1993

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93-31330



United States Army
Belvoir Research, Development and Engineering Center
Fort Belvoir, Virginia 22060-5606

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REPORT DOCUMENTATION PAGE**Form Approved**
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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE October 1993	3. REPORT TYPE AND DATES COVERED Final	
4. TITLE AND SUBTITLE Investigation of the Ultrafiltration Technique Using Military Greases (U)			5. FUNDING NUMBERS A391A207241	
6. AUTHOR(S) In-Sik Rhee				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Belvoir Research, Development & Engineering Center Logistics Equipment Directorate, Fuels and Lubricants Division Fort Belvoir, VA 22060-5606			8. PERFORMING ORGANIZATION REPORT NUMBER 2544	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) US Army In-House Laboratory Independent Research (ILIR) Project			10. SPONSORING/MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES POC: In-Sik Rhee (703) 704-1824				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Distribution unlimited; approved for public release.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) <p>Solid particle contamination in a grease intended for lubrication of instrument precision bearings can seriously degrade grease performance and bearing life. To produce clean grease, an ultrafiltration technique was recently introduced into the instrument bearing community. A major potential benefit by using this technique is that many instrument greases can be remanufactured from a variety of readily available lubricating greases. To assess this technique, an investigation was conducted to determine (1) whether the physical and chemical properties of filtered greases are altered by the filtration process, and (2) whether the filtered greases can improve instrument bearing life and their tribology properties. Results indicated that ultrafiltration can be performed on various types of greases with a substantial improvement in the resultant cleanliness level. However, some greases with rigid, discrete thickeners, and solid additives are unlikely candidates for ultrafiltration due to their physical characteristic changes after filtered.</p>				
14. SUBJECT TERMS lubricating greases grease filtration technique precision instrument bearings			15. NUMBER OF PAGES 23	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT Unlimited	

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Section I

Background

Precision instrument bearings are currently used in aircraft avionics and aircraft/missile guidance systems. To maintain the high precision, low friction, and high reliability, that operational requirements have imposed demands "ultraclean" greases specially engineered for these applications. Currently, two military instrument bearing grease specifications are widely used for these applications. One is MIL-G-23827, *Grease, Aircraft and Instrument, Gear and Actuator Screw*, and the other is MIL-G-81937, *Grease, Instrument, Ultra Clean*. Both greases are formulated with diester oil, lithium thickener, and additives. In an informal survey conducted by the Department of Defense (DoD) Instrument Bearing Working Group (IBWG), these greases did not provide good service life in high temperature applications and often gave inadequate corrosion protection to bearing materials.¹ For these reasons, there is a great need for new "ultraclean" instrument bearing greases to improve the life of bearing systems.

Most military instrument bearing lubricating greases are formulated for intended use in extreme field environments and multipurpose applications. To meet these requirements, the following physical and chemical properties are generally required in military instrument bearing grease specifications.

- Wide operational temperature ranges (-54 °C to 121 °C)
- Low friction coefficient and initial torque
- Excellent corrosion protection
- Wear preventive characteristics
- Quiet service
- Long operational and storage life
- Ultraclean

Ultraclean grease is required to avoid the severe wear problem of small precision bearings and the degradation of bearing life. Solid particles or dirt contaminants in grease usually originate from raw materials and manufacturing process or environments.² Normally, these particles are controlled during grease manufacture.

Today however, many suppliers in the grease industry are not willing to produce instrument bearing greases because of the high cost and difficulty in the manufacturing process. Therefore, only a few precision bearing greases are available to the instrument bearing community.

To increase the availability of "ultraclean" greases, an ultrafiltration technique was recently introduced into the instrument bearing community. A major potential benefit by using this technique is that many instrument bearing greases can be remanufactured from a variety of readily available petroleum and synthetic-based bearing greases. On the other hand, many workers in the grease industry are concerned that the ultrafiltration process may remove critical additives from some greases or change the structure of the thickener, adversely affecting their performance. Unlike oils, which contain soluble additives, greases are formulated with finely divided insoluble thickeners, and may also contain insoluble additives that may or may not be removed by ultrafiltration process.

To resolve this concern, a project was recently initiated to determine (1) whether the physical and chemical properties of filtered greases are altered by the filtration process, and (2) whether the filtered greases can improve instrument bearing life.

Section II

Evaluation of Ultrafiltration Procedures

The primary task of any filter is to remove and retain some unwanted substance from a useful medium. In grease filtration, the unwanted substance is usually defined as the *particulate contamination*. Unlike oils, the grease filtration can not normally be performed using the general laboratory equipment due to the limited gravimetric flow of grease. For this reason, grease filtration is always performed using high-pressurized equipment such as a hydraulic system or high pressure-pump which has the capability to develop the necessary pressures to force the grease through the supported membrane. Using this type of equipment, the lubricating greases are currently ultrafiltered at pressures below 6,800 KPa using membranes in the 3, 5, 10 micron pore size range.

The most important step in the filtration process is to determine the residual particulate contamination in the filtered grease. The contamination level of grease is commonly described by the number of particles within specified size ranges in defined area.³ Particle sizes are determined by the largest dimension in micrometers and its counting is usually done under microscope. In military grease systems, cleanliness has been classified into three contamination levels; regular, clean, and ultra-clean. A grease containing a particle size larger than 75 micron is defined as normal or regular grease, while the other is considered a clean grease. Typically, a grease having a particle size less than 35 micron is currently considered an ultraclean grease. This classification has been used for a long time in several military grease specifications to control particulate contamination. Table 1 shows the military specification requirements for particulate contamination level.

Table 1. Military Specification Requirements for Particulate Level

MICRON SIZE	MIL-G-81322D	MIL-G-23827	MIL-G-81937A
10 or larger			1,000
35 or larger			0
25 to 74	1,000	1,000	
75 or larger	0	0	

MIL-G-23827: Grease, Aircraft and Instrument Gear and Actuator

MIL-G-81937A: Grease, Instrument, Ultra-clean

MIL-G-81322D: Grease, Aircraft, General Purpose, Wide Temperature Range

Section III

Approach

The investigation of ultrafiltration technique was directed toward comparing the physical and chemical properties of filtered and unfiltered greases. Initially, two military greases were selected for this study. One was MIL-G-81322D, *Grease, Aircraft, General Purpose, Wide Temperature Range* and the other was MIL-G-10924F, *Grease, Automotive and Artillery*. Both greases were not originally formulated for use in instrument bearing applications. MIL-G-10924F grease was designed for ground vehicle applications while MIL-G-81322D grease was formulated for aviation applications. These greases have a technological similarity in that they both use similar synthetic base oils that impart excellent wide temperature properties as well as thermal and oxidation stability to the grease. They differ, however, in their thickener systems and additive chemistries to meet the performance requirements of their respective specifications. Typically, MIL-G-10924F grease was designed to provide saltwater corrosion protection while MIL-G-81322D grease provides fresh water corrosion protection. Their major differences in the physical and chemical properties are listed in Table 2.

Table 2. Physical and Chemical Properties of Test Greases

	MIL-G-81322D	MIL-G-10924F
Base Oil	PAO*	PAO+ Mineral Oil
Thickener	Clay	Lithium Complex
NLGI Consistency Number	1	2
Operation Temperature	-54 TO 180 °C	-54 TO 180 °C
Corrosion Protection	Freshwater	Saltwater

*Polyalphaolefin

To prepare the test samples, the greases selected for this study were ultrafiltered using ten micron pore size membranes. A device utilized in this filtration study was the NYE grease filtration equipment which is shown in Figure 1. The ultrafiltration procedure basically consists of charging the hollow stainless steel cylinder with the lubricating grease to be filtered and activating the pneumatic ram to discharge the grease through the filter assembly. The particulate contamination level of filtered

greases were determined by Federal test method (FTM) 791B. 3005.4, *Dirt Content of Grease*. The method consists of placing a minute amount of grease onto a thin template (20mm x 10mm x 0.1mm) contained between cleaned glass slides, and examining the slide under a microscope to determine the size and number of particles in the lubricating greases.



Figure 1. NYE Grease Filtration Equipment

To determine solid composition of filtered greases, a thermogravimetric analysis (TGA) method⁴ was adopted in this study. This method was originally developed to measure volatility of lubricants and residue. In this TGA analysis, only base oil and soluble additives of lubricating grease evaporate through a wide temperature range (up to 500 °C), while the insoluble additives and thickener system remain as the residue of sample. The results obtained from the baseline tests, which was conducted to verify this technique, agreed with the original formulation. Therefore, it was

concluded that this technique can be effectively used to analyze the amounts of solid composition in lubricating greases as a screening test. A typical TGA output is shown in Figure 2.

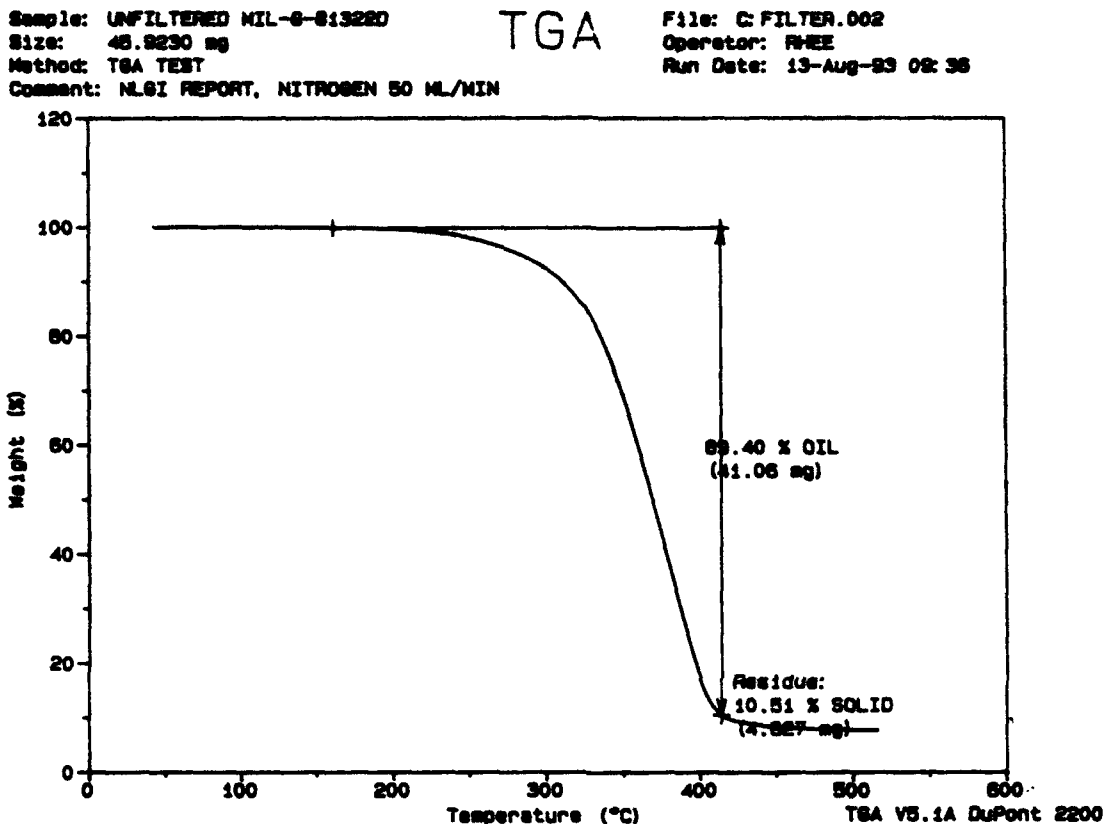


Figure 2. Typical TGA Thermograph

To assess the ultrafiltration technique, physical and performance tests were conducted to compare the properties of filtered and unfiltered greases. The test methods used in this investigation were the grease qualification test methods that are currently utilized in military specification MIL-G-10924F. Data from unfiltered greases were used as a baseline for this study. The tribology properties of the test samples were determined by the bearing life and wear tests. To determine oxidation life of the filtered greases, the Pressure Differential Scanning Calorimeter (PDSC) method⁵ was used. Noise tests were also conducted to compare noise levels of precision bearings lubricated with and without filtered greases.

Section IV

Discussion of Test Results

Ultrafiltration results are shown in Table 3 with photomicrographs (x250) displayed in Figure 3. No particles larger than 35 micron size were found in either filtered sample. Table 4 shows the total solid composition of test greases which was measured at before and after filtration using TGA analysis. In this TGA analysis, about one percent of the solid composition was filtered out of the MIL-G-81322D grease, while only 0.4 percent of solid was removed from MIL-G-10924F grease. It appears that the ultrafiltration process not only removes particulate contamination, but also filters out solid materials such as thickener or insoluble additives.

Table 3. Particulate Contamination Level for Test Greases

MICRON SIZE	MIL-G-81322D		MIL-G-10924F	
	Unfiltered	Filtered	Unfiltered	Filtered
10 to 34	300	0	250	0
> 35	200	0	250	0

Table 4. Total Solid Composition of Test Greases from TGA*

	UNFILTERED	FILTERED	CHANGES
MIL-G-81322D	10.51%	9.598%	0.912%
MIL-G-10924F	16.75%	16.36%	0.39%

* Thermogravimetric Analysis

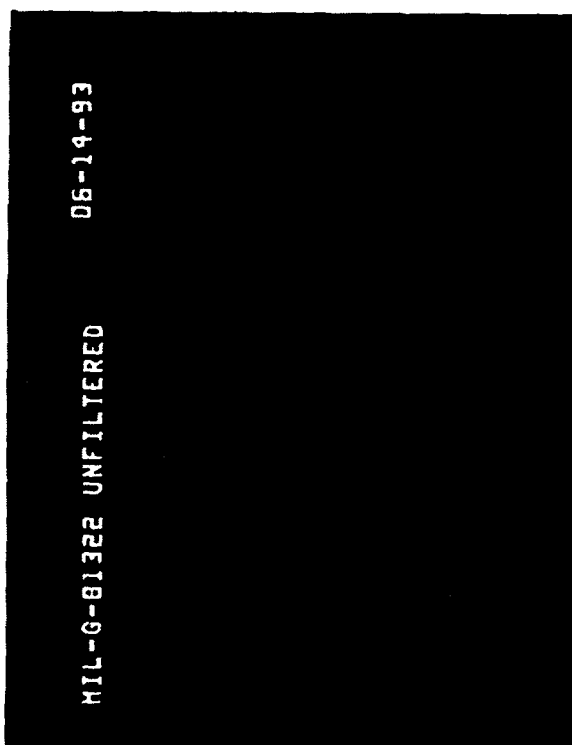


Figure 3. Microphotograph of Test Greases (x250)

A summary of the laboratory test results is presented in Table 5. The data obtained from each filtered and unfiltered grease were generated from grease performance test methods used in the MIL-G-10924F specification, FAG noise test method, and in-house triology test methods (SRV, Torque test, PDSC, etc.) for instrument bearing greases. Table 5 shows that both filtered greases provided very similar results in comparison with those of unfiltered greases except for the corrosion test. In the distilled water corrosion test, it was found that the ultrafiltration process removed crucial additives from MIL-G-81322D grease and adversely affected corrosion protection property. It appears that the sodium nitrate used for rust inhibitor in this formulation was apparently removed by the ultrafiltration process. In fact, the sodium nitrate is a fine solid powder and its solubility is very low in base oil. Unlike the MIL-G-81322D grease, the MIL-G-10924F grease passed both distilled water and saltwater corrosion tests. These results imply that grease formulated with insoluble inhibitors has a greater chance to have its performance adversely affected by ultrafiltration. Figures 4 and 5 show three bearing raceways tested with filtered and unfiltered grease samples in both distilled water and saltwater.

A primary concern regarding the production of ultraclean grease through filtration is the removal of thickener, which consist of extremely fine particles, with solid particulate. In grease formulation, the types of thickener material and its quantity are vitally important to obtain a stable grease structure and physical property. The unbalance of thickener ratio with base oils is evident in their consistency stability, mechanical stability, excessive oil separation, and thermal-oxidation stability. If the thickener ratio is decreased due to the ultrafiltration, the filtered grease may undergo softening in penetration due to the grease structure changes or a significant decrease in the high temperature expectancy. In these tests, the consistency stability of tested greases were determined by the worked penetration test. The test results indicated that both of the filtered greases do not alter in the penetration determinations from unfiltered greases.

To evaluate the mechanical stability of greases, the MIL-G-10924F specification requires the worked stability test and roll stability test. Both tests were designed to measure the penetration changes in consistency due to the continuous application of shearing forces. If filtered grease have a mechanical stability problem, they usually appear normal before being subjected to service but will soften rapidly upon working due to the low concentration of thickener in grease. These results directly depend on the ultrafilterability of grease. In these tests, both filtered greases did not show any abnormal behavior.

Table 5. Laboratory Test Results

TEST METHOD		MIL-G-81322D		MIL-G-10024F	
		Unfiltered Grease	Filtered Grease	Unfiltered Grease	Filtered Grease
Dropping Point, °C	ASTM D2265	+343	+343	262	260
Worked Penetration	ASTM D217	314	307	287	286
Work Stability, 100,000 Double Stroke	ASTM D217	15	8	29	25
Roll Stability	ASTM D1831	10	7	9	11
Evaporation, %	ASTM D972	1.0	1.0	2.0	2.1
Oil Separation, %	ASTM D1742	0	0	0	0
Centrifuge, Oil Separation, 2 hr, 40 °C, %	Modified ASTM D4425	13.5	15.0	6.9	8.2
Four Ball EP, LWI	ASTM D2596	35	34	30	30
Four Ball Wear Scar Dia., mm: Friction Coef:	ASTM D2264	0.67 0.1	0.62 0.1	0.53 0.1	0.5 0.08
SRV Wear Scar Dia., mm: Friction Coef: Step Load, N:	ASTM Draft Method	0.24 0.1 500	0.24 0.1 500	0.2 0.12 1,000	0.18 0.1 1,300
Copper Corrosion	ASTM D4048	1B	1B	1A	1A
Distilled Water Corrosion	ASTM D1743	PASS	FAIL	PASS	PASS
Saltwater Corrosion, 1% NaCl	Modified ASTM D1743	FAIL	FAIL	PASS	PASS
Low Temperature Torque @ -54 °C, N.m Breakaway: Running:	US Army Torque Test	3.6 1.72	3.7 1.7	4.9 2.87	4.7 2.8
Bearing Torque @ 22 °C, N.m Breakaway: PDSC*, @ 210 °C, min	US Army Torque Test	0.44 35.6	0.32 31.4	0.34 18.3	0.34 17.4
Noise Level	FAG Test	> VI/2	IV/2	III/3	III/3
Bearing Life, hr 160 °C: 130 °C:	ASTM D3527	200 730	200 620	100 1,000	100 >1,200

* Pressure Differential Scanning Calorimeter (PDSC)

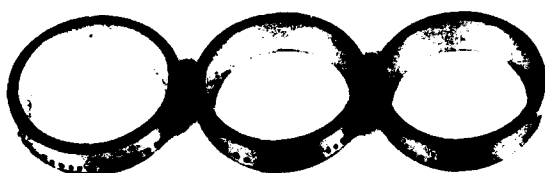


Unfiltered Grease



Filtered Grease

MIL-G-10924F



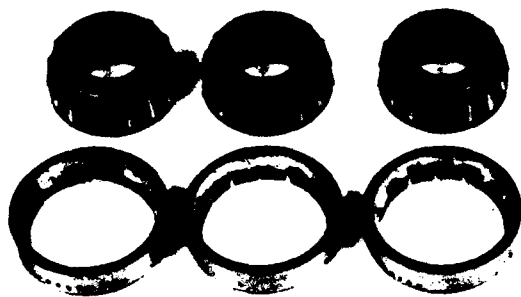
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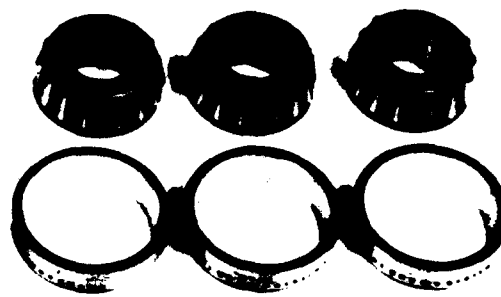
Filtered Grease

MIL-G-81322D

Figure 4. Distilled Water Corrosion Test



MIL-G-81322D



MIL-G-10924F



Unfiltered Grease



Filtered Grease

MIL-G-10924F

Figure 5. Saltwater Corrosion Test (1% NaCl) Results

Excessive oil separation of filtered grease often indicates that the significant amount of thickener is being removed through ultrafiltration. To assess this physical property, a static oil separation test was conducted according to the ASTM D1742 Method, *Oil Separation from Lubricating Grease During Storage*. The results did not indicate any oil separation from the both filtered and unfiltered greases. Dynamic oil separation tests were conducted using the modified ASTM D4534 Method, *Oil Separation from Lubricating Grease by Centrifuging (Koppers Method)* to verify these results. The results showed that both filtered greases gave a slightly high oil separation when compared to those of unfiltered greases. It appears that there was a removal of thickener by ultrafiltration.

The thermal-oxidation stability of the filtered greases was comprehensively evaluated using the results obtained from the dropping point test, evaporation test, and the Pressure Differential Scanning Calorimetry (PDSC) test. If the ratio of thickener and base oil in filtered greases is changed due to the ultrafiltration, the dropping point and evaporation of greases would also be expected to be adversely affected. Fortunately, we did not observe this type of problem in both filtered greases. To evaluate oxidative life of filtered greases, the oxidation tests were conducted using the proposed ASTM PDSC method. This method is currently being developed to assess oxidation stability of the lubricating greases using the differential heat flow between sample and reference thermocouple at various temperatures (155 °C, 180 °C, 210 °C) under pressure, 3.5 MPa. In this procedure, the degree of oxidation stability at a given temperature is determined by an induction time. The PDSC test results showed that the induction times of filtered greases are not affected by ultrafiltration. It appears that liquified or oil-soluble inhibitors may not be filtered out through ultrafiltration.

Tribology properties (friction, wear, lubrication) are one of important operational parameters in instrument bearing greases. Most precision bearing lubricants often use anti-wear additives to improve their wear prevention property. This property is usually evaluated in the Four Ball Wear test. To determine whether clean grease can improve wear prevention on the bearing surface, the four ball wear tests were performed according to the ASTM D2266, *Wear Preventive Characteristics of Lubricating Grease (Four-Ball Method)*. The test results indicated that the filtered greases provided a better wear protection on bearing surface than those of unfiltered greases. These results were also confirmed in SRV wear tests.⁶ It clearly showed that solid particles can cause very serious damage to precision bearings, either long-term wear by fine particles or rapid scoring by coarse particles. Unlike wear properties, the extreme pressure properties, low temperature torque values, and friction coefficient of filtered greases did not change due to the ultrafiltration. Table 5 identified torque test data obtained at room temperature. It showed that the

filtered MIL-G-81322 grease improved its initial torque value while the filtered MIL-G-10924F grease was unchanged. These results imply that ultrafiltration can improve the tribology properties of lubricating greases.

A high level of noise generated from the instrument bearings is usually caused from the surface defects or damage of the anti-friction components (ball, races), or due to the solid or semi-solid particles of the lubricant (dust, crystallized inhibitors, etc.).⁷ To assess noise levels of tested samples, noise tests were conducted using a FAG noise test rig MGG 11. Table 5 showed that the filtered MIL-G-10924F grease did not improve the quality of noise level while filtration improved the noise level of MIL-G-81322D grease. Homogeneity and cleanliness resulted from the removal of large size clay particles or solid additives formulated in MIL-G-81322D grease, slightly contributed to the reduction of noise level.

Clean greases tend to improve the bearing life.⁸ To verify this result with filtered greases, bearing tests were conducted according to the ASTM D3527 Method, *Life Performance Test of Lubricating Greases*. In this test, bearing life is represented by the grease life. Table 5 showed that the filtered greases did not improve the bearing life in the high temperature bearing tests (160 °C). To clarify this area, the bearing tests were performed again at the maximum operating temperature (130 °C) of instrument bearings. In these tests, the filtered MIL-G-10924 grease improved bearing life while the filtered MIL-G-81322D actually reduced bearing life. It appears that ultrafiltration process adversely affected the performance of MIL-G-81322D grease. On the other hand, MIL-G-10924F grease tended to improve the bearing life at the operating temperature of instrument bearings. It also found that the bearing life data obtained at 160 °C were not correlated to those at the lower temperature (130 °C). It appears that the high temperature life of grease totally depends on their thermal stability rather than their cleanliness. Evidently, the life of unfiltered MIL-G-10924F grease was better than that of MIL-G-81322D at 130 °C while their life were opposite at the high temperature (160 °C).

Section V

Conclusions

Based on our limited investigation on the grease ultrafiltration technique, it can be concluded that this technique may be useful to produce clean greases from a limited variety of readily available petroleum and synthetic-based lubricating greases. Also, the bearing life and its tribology properties can be improved using filtered greases. The results of this study are summarized in the following findings.

1. Lubricating greases can be ultrafiltered to reduce particle sizes below 35 microns, and to minimize the number of particles between 10 and 35 microns.
2. The corrosion test results showed that the filtered MIL-G-81322D grease failed distilled water corrosion test due to the removal of a solid corrosion inhibitor by filtration.
3. TGA results showed that a significant amounts of solid particles (i.e, thickener or additives) were removed from MIL-G-81322D grease.
4. Clean grease can improve the instrument bearing life and its tribology properties. A high temperature bearing life totally depends on the thermal stability of lubricant rather than the clean grease.
5. Noise level may or may not be reduced by the filtered greases.
6. MIL-G-10924F grease was not significantly changed its performance after filtered, but its cleanliness level was improved.

Therefore, it is recommend that filtered greases must be re-tested prior to use in the instrument bearings because some filtered greases may alter initial performances.

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